pH Determination of Mainstream Smoke

Objective: To measure the pH of mainstream cigarette smoke in an aqueous solution through the analysis of a combined sample of smoke collected in water (using glass impingers) and TPM (collected on a Cambridge filter pad). Cigarettes are smoked in accordance with Federal Trade Commission (FTC) guidelines using a Filtrona 20-port smoking machine.

Apparatus & Instrumentation

1. Accumet pH/ion meter, model 25, Cat. #13-635-25, Fisher Scientific.
2. Accumet glass body combination electrode, Ag/AgCl internal reference element, Cat. #13-620-285, Fisher Scientific.
3. Glass Impingers, (smoke collection traps), Research Glass.
4. Magnetic stir plate, Cat.# 27 25 001-7, Brinkmann.

Chemicals:

1. Buffer Solutions, pH 4.0 Cat.# SB101-500, pH 7.0 Cat.# SB107-500, and pH 10.0 Cat.# SB115-500, Fisher Scientific.
2. Hydrogen Peroxide 3%, Cat.# H324-500, Fisher Scientific.
3. Degassed Deionized Water, 18 megaohms-cm, (water is supplied from a Milli-Q water system and degassed with helium).

Preparation for Analysis:

1. The pH electrode should be cleaned daily before calibrating the pH meter. 3% hydrogen peroxide solution is used for cleaning the pH electrode. To clean the electrode, place it in the 3% hydrogen peroxide solution for approximately 2 minutes then remove the electrode from the solution and rinse with Milli-Q water.

2. Calibration of the pH meter is performed daily before taking any pH measurements. The pH meter is calibrated using pH 4 and pH 10 buffers for a two point standardization. The electrode slope or efficiency must be within 90% to 105% before the pH meter can be used for sample measurements. The pH measurements are performed at controlled temperature and humidity conditions (75^0 F $\pm$ 2^0 F with 60 % $\pm$ 2 % humidity).

3. The electrode response should be checked against the pH 7 buffer before each impinger sample measurement. The buffer response must be within 7.00 $\pm$ 0.10 pH units or the pH meter must be recalibrated.

4. The smoke from one cigarette sample is collected using 2 glass impingers connected in series, and a Cambridge filter pad. 50.0 mL $\pm$ 0.2 mL of degassed Milli-Q water is used in each impinger. The Milli-Q water is degassed overnight for use that day. The Milli-Q water should be degassed no more than 12 hours before use.

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5. Samples must be analyzed within 10.00 ± 2.00 minutes after sample collection.

Test Procedure:

1. Remove the impingers from the sampling apparatus after the final clearing puff and start the timer.

2. Shake the traps vertically for ~20 seconds being careful not to spill any of the sample solution.

3. Transfer the contents of the two impingers and the Cambridge filter pad to a 150 mL beaker that contains a magnetic stir bar. (Note: Use finger cots when transferring the filter pads.)

4. Place the beaker on a magnetic stir plate and stir gently.

5. Submerse the pH electrode into the beaker being careful that the electrode does not come into contact with the filter pad or the stir bar.

6. Record the pH when the timer stops (which should be ten ± two minutes after the sample was collected).

Note: Between sample and standard measurements the electrode should rest in deionized water. Before taking the next reading, rinse the electrode with deionized water and blot dry with a tissue (Kimwipe) to prevent sample contamination.

Analysis Order:

1. Calibrate the pH meter using pH 4 and pH 10 buffers daily.

2. **Perform the calibration standard check with pH 7 buffer before each impinger measurement.**

3. Measure the pH of the sample blank (using IM 15 or 16 - perform the entire test procedure without lighting the cigarette).

4. Measure the pH of IM #15.

5. Measure the pH of IM #16.

6. Measure the pH of the test samples.

7. Perform the calibration standard check with pH 7 buffer.

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Note: Dispose of all chemicals in accordance with R&D policy.

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